STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF
WATER CONTENT BY KARL FISCHER TITRATION
(Based on SW846 Method 9000)

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1. SCOPE AND APPLICATION:

    1.1. This method is applicable to the determination of water content of paint, varnish, lacquer or related surface coatings, and wastes. It is used to determine water content under 40 CFR Part 60 Appendix A (Method 24 and 24A), and to support RCRA characteristic testing under 40 CFR part 261.21 and 261.22.

    1.2. This method does not lend itself to method detection limit (MDL) determinations.

    1.3. The method determines water in concentration ranges from 0.1 – 100 %.

        1.3.1. The Reporting Limit (RL) for water content determined by this SOP is set at 0.5 %.

        1.3.2. LIMS Instrument ID: KFT.

    1.4. The estimate of minimum laboratory contribution to measurement uncertainty of this method for water content analysis is 102 ± 5 % Recovery (mean ± 3 std dev). This value is derived from CRL historical data for Laboratory Control Samples (LCS) for this method. The LCS is a manufactured water content standard at 10.0 mg H_2O/mL. The uncertainty will be greater near the reporting limit and much greater near the detection limit.

    1.5. See Appendix C for deviations from the reference method.

2. SUMMARY OF METHOD:

    2.1. A sample aliquot is dissolved in a suitable solvent and titrated with standardized reagent to an electrometric end point.

3. ABBREVIATIONS AND DEFINITIONS:

    3.1. CALIBRATION STANDARD (CAL) – A standard used to calibrate the instrument with respect to water concentration.

    3.2. QUALITY CONTROL SAMPLE (QCS) – A solution of known water content obtained from a source external to the laboratory. The solution is used to check laboratory performance with externally prepared test materials.

    3.3. LABORATORY DUPLICATES (DUP) – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analysis of a number of sample and DUP indicates precision associated with the laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.4. FIELD DUPLICATE (FD1 and FD2) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analysis of a number of FD1 and FD2 indicates the precision associated with the sample collection, preservation and storage, but not with the laboratory procedures.

3.5. SAFETY DATA SHEET (SDS) – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

4. HEALTH, SAFETY AND WASTE HANDLING:

4.1. Users of this method should operate a formal safety program. Perform this procedure in accordance with the CRL Chemical Hygiene Plan located in CRL's (G) share drive.

4.2. Review SDSs for specific physical and health and hazards including appropriate PPE to be used. SDSs may be accessed at: www.sigmaaldrich.com. Alternatively other vendor websites may be used to locate pertinent information.

4.3. Safe laboratory procedures should be followed at all times.

4.4. Disposable syringes should be collected in hazardous waste containers following the laboratory Chemical Hygiene Plan.

4.5. **WARNING:** The reagents used in this procedure contain toxic compounds and should be handled with care. Care must be exercised to avoid inhalation or skin contact.

4.6. Report all major spills and injuries.

4.7. Dispose of reagent wastes into green labeled waste containers.

4.8. All other wastes produced in performing this method must be disposed of according to the Chicago Regional Laboratory Chemical Hygiene Plan.

5. CAUTIONS AND INTERFERENCES:

5.1. The possibility of interfering side reactions or the formation of byproducts, which may prevent accurate determination of water content, may occur. This method has been developed to minimize those potential problems but if they are suspected, it should be reported.

5.2. It is essential to use care and follow precise procedures when dissolving and mixing the specimen samples to obtain a homogeneous and therefore representative sample of the coating, ink, or waste.

5.3. All reagents used should be less than one year old, or within the manufacturer's expiration date, and from a reliable laboratory reagent supplier. Contamination of reagents and solutions may cause significant reproducibility problems.

6. EQUIPMENT AND SUPPLIES[1,2]:

6.1. Calibrated Support Equipment:

6.1.1. Analytical balance – capable of weighing to 0.0001 g. Certified yearly by vendor or an outside source.

6.1.2. Top loading balance – capable of weighing to 0.01 g. Certified yearly by vendor or an outside source.

6.1.3. Weight set covering the range of balance use, which is typically up to 100 g. Rice Lake Weighing systems (or equivalent). Certified yearly by vendor or an outside source.

6.2. Titration Apparatus:

6.2.1. Metrohm Automatic Karl Fischer Apparatus (or equivalent).

6.2.1.1. LIMS ID: KFT

6.2.2. Tiamo Titration Manager (or equivalent).

6.2.3. Metrohm Titrando #901 (or equivalent).

6.2.4. Metrohm Dosino Dosing unit #800 (or equivalent).

[1] Refer to CRL SOP GEN026 for instructions on purchasing equipment and supplies.
[2] The following brand names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

6.3. 10 mL disposable syringe without needle, but equipped with a cap.

6.4. Hamilton Gastight Syringes: 100 µL (#1710), 1.0 mL (#1001), 5.0 mL (#1005), 10 mL (#1010). Or equivalent.

6.5. Data System.

7. REAGENTS AND STANDARDS[1,2]:

7.1. Reagent water – ASTM type II water (or equivalent).

7.2. Hydranal–Composite 5K, Fluka Analytical Part # 34816 reagent grade (or equivalent).

7.3. Hydranal–Working Medium K, Fluka Analytical Part # 34817 reagent grade (or equivalent).

7.4. Hydranal-Water Standard 10.0, Fluka Analytical Part # 34849, commercially certified standard (or equivalent).

7.5. Prior to preparation of reagents and standards, refer to Appendix B for directions on record keeping and appropriate dissolving, mixing, and labeling methods.

7.5.1. To prevent materials from degradation follow the manufacturer's recommendations for preparation, handling, and storage.

8. SAMPLE HANDLING AND PRESERVATION:

8.1. Paint and coating samples:

8.1.1. Coating samples are collected in cans or containers suitable for paints and solvents.

8.1.2. The sample container should be filled to overflowing prior to placing inner seal in container. A partially filled container indicates potential VOC loss or some other problem with the sample. A repeat sample is required.

8.1.3. Samples should be delivered to the laboratory for analysis the same day they were collected.

8.1.4. Maintain samples at room temperature, preferably at 70 °F but within the range of 40 °F to 100 °F.

8.1.5. Coatings with relatively high vapor pressures (those containing volatile solvents with boiling points below 100 °F) should be packed in ice to keep them within the range of 40 °F to 100 °F.

8.1.6. Analyses should begin as soon as possible. If not possible all sample analyses must be completed within 30 days. If the analyses have not been completed within 30 days, it is recommended that another sample should be collected. Once the sample jar is open the analyses must be completed within 48 hours.

8.2. RCRA samples:

8.2.1. RCRA characteristic samples may be collected in glass jars or other appropriate containers.

8.3. Upon receipt, samples should be verified for proper preservation. If any sample is found to be improperly preserved, the customer must be notified in order to determine the correct action.

8.4. If there are problems associated with sample collection, storage, or integrity the sample coordinator and customer service liaison must be contacted and informed of the specific issues.

9. SAMPLE PREPARATION AND ANALYSIS:

9.1. Check the balance daily with a weight set covering the range of measurement. See Section 6.1 for analytical balance requirements.

9.2. *Standardization of Karl Fischer Reagents*:

9.2.1. To start the software, click on the **Titrando** icon.

9.2.2. Prepare the dosing unit to rinse and fill the cylinder and tubing so that they are free of air bubbles. You should carry out this function before the first determination or once per day. Proceed as follows:

9.2.2.1. In the sidebar, click on the symbol **Manual Control**.

9.2.2.2. In the left hand window, select the dosing device to be prepared.

9.2.2.3. Click on the tab **Prepare.**

9.2.2.4. Click **Start** and confirm the message with **Yes.** Preparation is carried out for the dosing unit.

9.2.2.5. Close the manual control after the preparation has been completed. Click on **Close**.

9.3. *Titration of Reagent Water to Determine the Concentration of the Titre:*

9.3.1. In the sidebar, click on the symbol **Workplace**.

9.3.2. In the sub window **Run** under **Method**, select the method **KF TITRE.**

9.3.2.1. **Note:** The method **KF TITRE** has already been configured and optimized. Do not edit the method without consulting the Primary Analyst or Group Leader. When the method does require updating make note in the instrument logbook and archive the method with the data per Section 9.7. Since the Primary Analyst and/or Group Leader has already been notified they will inform SOP users and/or update the SOP as necessary.

9.3.3. In the **Sample ID** field, enter **Water**.

9.3.4. In the **Water Standard ID** field, enter **1000.**

9.3.5. Begin the pre-titration by clicking on **Start.** The instrument will automatically initiate titration of the solvent to eliminate any moisture it contains and indicates when the pre-titration of the solvent is completed.

9.3.6. While the pre-titration is in progress, fill the syringe with water to at least the 1 mL mark. Place the syringe on the balance and press tare on the balance.

9.3.7. After pre-titration is completed, a **Conditioning Ok** message appears. Click on **Start** and inject approximately 4 drops of water from the syringe into the titration vessel using the sample port. Immediately replace the sample port stopper to reseal the titration vessel.

9.3.8. Place the syringe on the balance and press Print on the balance. The sample mass will be automatically entered into the **Sample Size** field.

9.3.9. Titration will end when the determination is completed. A sub window will appear with the calculated result of the titre. The instrument

automatically calculates the Karl Fischer Titre (refer to Section 11 for the calculation formula).

9.3.10. Repeat the standardization until replicate values of the titre agree to within 1%. The software will automatically save the concentration of the titre.

9.4. *Standardization Verification with Reference Material (SRM):*

9.4.1. In the sidebar, click on the symbol **Workplace**.

9.4.2. In the sub window **Run** under **Method**, select the method **KF Sample.**

9.4.2.1. **Note:** The method **KF Sample** has already been configured and optimized. Do not edit the method without consulting the Primary Analyst or Group Leader. When the method does require updating make note in the instrument logbook and archive the method with the data per Section 9.7. Since the Primary Analyst and/or Group Leader has already been notified they will inform SOP users and/or update the SOP as necessary.

9.4.3. In the **Sample ID** field, enter **Hydranal 10.0.**

9.4.4. Begin the pre-titration by clicking on **Start.**

9.4.5. While the pre-titration is in progress, fill the syringe with the Hydranal 10.0 standard to at least the 1 mL mark. Place the syringe on the balance and press tare on the balance.

9.4.6. After pre-titration is completed, a **Conditioning Ok** message appears. Click on **Start** and inject 1 mL of the standard from the syringe into the titration vessel using the sample port. Immediately replace the sample port stopper to reseal the titration vessel.

9.4.7. Place the syringe on the balance and press Print on the balance. The sample mass will be automatically entered into the **Sample Size** field.

9.4.8. Titration will end when the determination is completed.

9.4.9. The concentration will be calculated automatically and will appear in the result sub window of the software.

9.4.10. Evaluate the result as recovery. Calculate recovery using the equation:

$$R = \frac{C_S}{C_T} \times 100\ \%$$

Where:

R = Percent Recovery (%).
C_S = Concentration of standard found (mg/mL).
C_T = Target concentration of standard (mg/mL).

9.4.10.1. Evaluate the recovery using the current limit in Section 10.6.

9.4.10.2. If the recovery exceeds the current limit and results are to be reported, the Group leader will be notified with the analyst's recommendation for a final evaluation of the data.

9.5. *SAMPLE ANALYSIS:*

9.5.1. Sample analysis:

TABLE 1: SAMPLE SPECIMEN GUIDE

Expected Water (%)	Approximate Specimen Weight (g)	Approximate Titrant Volume at 5 mg/mL titre (mL)
0.5-1.0	5	5-10
1-3	2-5	10-20
3-10	1-2	10-20
10-30	0.4-1.0	20-25
30-70	0.1-0.4	15-25
> 70	0.1	20

9.5.2. In the sidebar, click on the symbol **Workplace**.

9.5.3. In the sub window **Run** under **Method**, select the method **KF Sample.**

9.5.4. Begin the pre-titration by clicking on **Start.**

9.5.5. While the pre-titration is in progress, homogenize the sample.

9.5.6. Draw a sample using a syringe applying the specimen size guidelines given in Table 1 above. Remove the syringe from the sample container, pull the plunger out a little further, and wipe the excess material off the syringe.

9.5.7. Place the syringe on the balance and press tare on the balance.

9.5.8. After pre-titration is completed, a **Conditioning Ok** message appears. Click on **Start** and inject the sample from the syringe into the titration vessel using the sample port. Immediately replace the sample port stopper to reseal the titration vessel.

9.5.9. Place the syringe on the balance and press Print on the balance. The sample mass will be automatically entered into the **Sample Size** field.

9.5.10. Titration will end when the determination is completed.

9.5.11. The software will automatically calculate the % water content of the sample and will appear in the result sub window of the software.

9.5.12. Repeat the procedure with another aliquot of the same sample.

9.5.13. Evaluate the difference between the two determinations using the duplicate difference criteria in Section 10.6.

9.6. A report of analysis results can be generated and printed on the system printer.

9.6.1. In the sidebar, click on the symbol **Database**.

9.6.2. Select sample determinations as follows:

9.6.2.1. Place cursor in the column under a sample determination. Hold down the right mouse key and drag the mouse down over the desired samples. Unclick the mouse. Selected samples appear highlighted in blue.

9.6.2.2. From the menu, click on **File,** then **Print,** then **Report.** A new window will appear.

9.6.2.3. Under Selection, click on **Selected Determinations.** Under Report Type**, click on Original Report.** Under Output Target, select printer.

9.6.2.4. Click on **OK** to print report.

9.7. Data Backup permits the operator or manager to export sample data to a file. The file can be saved on the systems hard drive or transferred to another media for safe storage. If necessary, the file can be imported and the sample data restored.

9.7.1. In the sidebar, click on the symbol **Database**.

9.7.2. Click and drag the mouse pointer to select the rows with sample determinations to export to a file.

9.7.2.1. In the menu, click on **Determinations,** then **Export.** The export file selection screen will appear.

9.7.2.2. Under Selection, click on **All Selected Data Records.** Under Export Template, select **CRL_Export.**

9.7.2.3. Click on **OK** and the target directory will be shown. Enter a file name and click on **OK** to save the selected sample determinations to a file.

9.7.2.4. The saved file can now be uploaded to a network drive for archiving.

10. QUALITY CONTROL:

10.1. Users of this method must operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and analysis of QCS solutions as continuing checks on performance. The user is required to maintain performance records that define the quality of the data that are generated. All limits are to be based on historical data.

10.2. For information on formal corrective actions refer to the Corrective Actions section in the Chicago Regional Laboratory (CRL) Quality Management Plan (QMP). Immediate corrective actions for specific QC audits are discussed in the subsections of this section.

10.3. INITIAL DEMONSTRATION OF PERFORMANCE:

10.3.1. Prior to analyzing samples by this method, the following determinations and analyses must be successfully completed and documented as appropriate.

10.3.1.1. Karl Fisher (KF) Reagent Titre Determination:

See Section 9.3.

10.3.1.2. Analysis of a Quality Control Sample (QCS):
Analyze one QCS per batch. Evaluate the results as % Recovery using the limits in Section 10.6.

10.3.2. Maintain all determination and analysis information in LIMS and a data package file.

10.4. ASSESSING LABORATORY PERFORMANCE:

10.4.1. Instrument Performance Check (IPC) Solution:

10.4.1.1. Daily determine KF reagent titre and analyze a check standard to pass. Refer to Sections 9.3 and 9.4 for the procedures.

10.4.1.2. **Note:** this is considered the daily calibration for this procedure, and if successful the calibration is expected to last throughout the day it was determined.

10.5. ASSESSING ANALYTE RECOVERY AND DATA QUALITY:

10.5.1. Laboratory Duplicate (DUP):

10.5.1.1. All samples are analyzed and accepted upon meeting duplicate criteria.

10.5.1.2. Analyze the sample (S) and duplicate (DUP).

10.5.1.3. Calculate the Duplicate RPD by using the following equation:

$$\mathrm{RPD} = \frac{|\mathrm{S} - \mathrm{DUP}|}{((\mathrm{S} + \mathrm{DUP})/2)} \times 100\%$$

Where:
RPD = Relative percent difference.
S = Sample concentration (% water).
DUP = Sample duplicate concentration (% water).

10.5.1.4. Evaluate the duplicate difference using the limit in Section 10.6.

10.5.1.4.1. If the duplicate RPD% is within the limits, no further action is required.

10.5.1.4.2. If the RPD% is out of the limit and results are to be reported, the team leader will be notified with the analysts recommendation for a final evaluation of the data.

10.5.2. Matrix Spike (MS):

10.5.2.1. Not performed in this SOP. See Appendix C – Deviations from Reference Method.

10.6. QUALITY CONTROL SUMMARY:

AUDIT	FREQUENCY	LIMIT	ACTION
SRM/QCS	Once per batch, beginning of analysis	102 ± 5 %	Reviewed January 2017 (99%, 3σ)[3]
DUP	Per site, and once for each group of 20 samples or fewer collected	RPD ≤ 10 %	Reviewed January 2017[4]

11. DATA AND RECORDS MANAGEMENT:

11.1. See Appendix A for LIMS benchsheet and LIMS information.

11.2. Calculations are performed automatically by the instrument software. The calculations below are included for reference purposes.

11.3. *CALCULATIONS:*

11.3.1. Calculate Karl Fischer Titre in grams of water per milliliter of Karl Fischer Reagent used with the following formula:

$$F = \frac{J}{K}$$

Where:

F = Karl Fischer Titre (g/mL).
J = Water added (g).
K = Karl Fischer Reagent used (mL).

[3] Insufficient data was available during this review cycle to recalculate the SRM control limit. Limit will remain the same as previous SOP Versions..

[4] Insufficient data was available during this review cycle to calculate the RPD limit. The limit of less than or equal to 10% was set at the maximum allowed by the reference method (SW846 Method 9000 Revision 0 February 2007).

11.3.2. The Tiamo software calculates the % water content in samples. The following formula can be used:

$$\% \text{ Water} = \frac{K \times F \times 100}{W}$$

Where:

K = Karl Fischer Reagent used (mL).
F = Karl Fischer Titre (g/mL).
W = Weight of Sample (g).

11.4. Data is reported to a maximum of three significant figures and one decimal in percentage units (%) as follows:

0.X, X.X, XX.X [%]

11.5. Concentrations lower than 0.5 % will be reported as requested by the customer.

11.6. Raw data and bench sheets are to be submitted with the data package.

11.7. Any irregularities in labeling or preservation of samples, or unusual observations must be documented in a case narrative and brought to the attention of the data user.

11.8. All electronic records associated with any data package must be archived.

11.8.1. See Section 9.7 for data backup instructions.

12. TROUBLESHOOTING:

12.1. Contamination of the KFR burets, the reagent reservoir glassware, the titration vessel, and the coating of the sensing electrodes are all potential problems to inspect for and to prevent by performing periodic cleaning.

12.2. Clean the titration vessel. Do not use methanol or other solvents.

12.3. Maintain anhydrous conditions (dryness) in the titration vessel by checking that drying tubes are in good condition and are tightly connected. Replace desiccant when indicator color changes through half the tube.

12.4. Follow manufacturer's guidelines regarding electrode performance response and cleaning.

13. PREVENTIVE MAINTENANCE:

13.1. Preventative maintenance records and log book are kept with the instrument.

13.2. Please refer to the instrument instruction manual that is located by the instrument for routine preventive maintenance.

14. REFERENCES:

14.1. 40CFR, Part 60, Appendix A, Method 24 and 24A.

14.2. 40 CFR, Part 261.21 and 261.22, Subpart C, Identification and Listing of Hazardous Waste. Current as of 01/05/2017 (Accessed On-line 01/09/2017): http://www.ecfr.gov/cgi-bin/text-idx?SID=1f5c7c4104dc04c7f534940143d767e8&node=sp40.26.261.c&rgn=div6

14.3. Tiamo Software Instruction Manual.

14.4. U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards; Stationary Source Compliance Division, EPA- 340/1-91-011, September 1991.

14.5. “Test Methods for Evaluating Solid Wastes. Physical/ Chemical Methods”, EPA Publication SW-846, Update IV, Method 9000. Determination of Water in Waste Materials by Karl Fischer Titration. Revision 0, February 2007. Accessed On-line 01/09/2017: https://www.epa.gov/sites/production/files/2015-12/documents/9000.pdf.

15. REVISION HISTORY:

Version	Status	Location of Change History
4	R	Cover Page: Replaced L-A-B logo with ANAB logo.
#3	R	This SOP was revised for the 2017 cycle according to GEN006 V#4 and CRL CHKLST 001A (SOP Short Checklist) V#4.
		1.5: Add reference to Appendix C for deviations.
		9.3.2.1 & 9.4.2.1: Added instructions on instrument method updates.
		10.4.1.2: added how long an instrument calibration is expected to last.
		10.6: Updated/Reviewed QC Limits.
		14: Updated/Reviewed References.
		Qualtrax DRAFT Document Workflow ID# 9113.
Version #2	R	Entire Document: Minor grammatical, formatting, and/or editorial changes were made throughout this SOP revision. The significant changes to procedures and/or quality control are itemized below: Section 1.2: Removed statement on MDL studies since this SOP does not lend itself to MDL determinations. Section 4: Section 4: Added statement for adherence to CRL's Chemical Hygiene Plan and information on SDS's. Section 6.1.3: Added balance check weight set to Calibrated Support Equipment. Section 6.4: Moved syringe information from the Calibrated Support Equipment to Section 6.4. The mass of a sample analyzed is determined gravimetrically and not by the volume of the syringe, therefore, syringes for this SOP need not be certified. Section 7.5: Added statement on preparing reagents and standards and preventing degradation to materials. Section 10.2: Added language on formal and immediate corrective actions. Section 10.6: Reviewed and/or updated the QC Limits Summary and dates of action taken. Section 14: Updated and/or reviewed references. Removed citation for ASTM D4017-90. This SOP follows EPA Method 9000. Appendix B Section 1.1: Updated language for LIMS documentation of reagent preparations. Appendix C: Added Appendix C to document deviations in this SOP from the cited reference method.
0.2	R	Entire Document: Reorganized and renamed sections, including the addition/deletion of sections; reformatted to

		include subsection numbering system; revised with editorial changes to comply with CRLSOP GEN006 Revision 7.0. Edits to this version are too numerous to summarize here, see the hardcopy Track Changes Document filed with this revision. QC: QC Limits for DUP updated to 12 % RPD. QC Limits for LCS updated to 102 ± 5 %. Procedure: No procedural changes.

Version – Version number of this document
Status – I = Initial, R = Revision, or C = Cancelled

APPENDIX A – LIMS Entry and Reporting

1.1 CRL uses Element DataSystem (ElmNT). Refer to Element DataSystem, version 4.0, New User Tutorial (CequeLogic) and associated references as necessary.

1.2 ***Creating a bench sheet:***

1.2.1 Create a LIMS bench sheet (bch_C_AIG015_Karl_Fischer_Titration.rpt or equivalent).

1.2.2 Refer to Section 10 of this SOP for general QC requirements.

1.2.3 Make sure that the preparation date in LIMS bench sheet matches the actual preparation date on the laboratory bench sheet.

1.2.4 After selecting DONE, ElmNT automatically creates and saves a file for the bench sheet just prepared.

1.3 ***Data entry:***

1.3.1 From the ElmNT pull down menu, select Laboratory, Data Entry/Review, and the bench sheets created in section 1.2.

1.3.2 Data is entered manually. Enter the results in the column **Result** in mg/mL. For each result, enter the date of analysis in the column **Analyzed**.

1.3.3 When all data are entered, click the **Save** button on the top row. After saving, proceed to the Review page by clicking **Query** on the second row. Flags may be added at this stage, following the guidance given in SOP GEN015. Before review by the peer, the data must be locked, and the status should be updated to Analyzed.

1.4 ***Report Generation:***

1.4.1 Preparation of a draft report:

1.4.1.1 Ensure that all data are entered with the status of Analyzed.

1.4.1.2 From ElmNT pull down menu, select Project Management and then Reports.

1.4.1.3 Choose the work order number, analysis, and appropriate report format for “Water content, Karl Fisher Titration”. Select Draft report.

Note: This draft report need not be signed. It is only for the purpose of review.

1.4.1.4 Submit the draft report with the data package to a peer reviewer.

1.4.1.5 After completing the data review, the peer reviewer updates the status of the LIMS entries to Reviewed.

1.4.2 Preparation of a final report:

1.4.2.1 After the peer reviewer has updated the status of the LIMS entries to Reviewed, a final report may be generated.

1.4.2.2 Ensure that all data are now in Reviewed status. Refer to section 1.4.1.2 and 1.4.1.3 of this appendix for generation of the report. Select Final Report or Modified Final Report. All pages of the report and the transmittal form must be signed and dated by the analyst.

APPENDIX B – Preparation of Reagents and Standards

The following procedure should be practiced for all preparation of reagents and standards.

1. Reagent or Standard Records

    1.1. Reagent Records

        1.1.1. Use a reagent/standard preparation benchsheet from LIMS with "std_C_reagent_prep_bench_sheet_blank.rpt" OR "std_C_reagent_prep_bench_sheet_filled out.rpt" OR an equivalent format for documentation. Submit preparation benchsheets with the data they were prepared with.

        1.1.2. Enter all reagent/standard preparation information from the above benchsheet(s) into LIMS. LIMS will assign a number. Once a LIMS number is assigned print out a label to put on the reagent/standard container.

2. Dissolving and Mixing Reagents

    2.1. General Instructions:

        2.1.1. All reagents and standard are prepared in volumetric flasks. Deviations are given in the specific reagent preparation section.

        2.1.2. Reagents are not to be heated to dissolve unless specified in the appropriate section.

        2.1.3. Mixing to dissolve solid reagents during preparation can be accomplished manually or mechanically.

            2.1.3.1. Manually by shaking and swirling to dissolve the solid reagents or mix liquid reagents.

            2.1.3.2. Mechanically by using Teflon coated magnetic stir bar and stirring plate.

        2.1.4. It is recommended that solid reagents be dissolved mechanically. Refer to Section 2.1.3.2.

        2.1.5. When magnetic stir bars and stirring plates are used to dissolve reagents, it is necessary to follow specific procedures to place and remove a stir bar from a flask in which the reagent solution is prepared.

2.1.5.1. Refer to Section 2.2 to place a stir bar in the solvent and begin to dissolve solid reagent.

2.1.5.2. Refer to Section 2.4 to remove a stir bar from the dissolved reagent solution.

2.1.6. Liquid reagents can be mixed manually to dissolve. Refer to Section 2.1.3.1.

2.1.7. When all reagents are dissolved, fill the flask or container to required volume. This is usually the mark if volumetric flask were used.

2.1.8. Final mixing can be done by repeated inversion of the flask or container.

2.2. Placing a Teflon coated stir bar in the flask or container with solvent:

2.2.1. Obtain a clean volumetric flask or container. The size of the flask or container is recommended under each reagent preparation section.

2.2.2. Fill the volumetric flask or container with the minimum amounts of reagent water or solvent suggested under the preparation section for the reagent.

2.2.3. Gently drop a Teflon coated stir bar into the volumetric flask (2.2.2)

2.2.4. Place the flask (Section 2.2.3) on a stirring plate and begin to gently stir the reagent water or solvent.

2.3. Dissolving solid reagent:

2.3.1. While stirring the reagent water or solvent as described in Section 2.2.4, carefully transfer the amounts of reagents required into the flask. This is obtained from the specific reagent preparation section.

2.3.2. Continue to stir the reagent solution in Section 2.3.1. While still stirring, cautiously add more reagent water to the volumetric flask until it is approximately 95 % full.

2.3.3. Remove the stir bar from the solution following the procedure given in Section 2.4.

2.4. Removing the stir bar from the reagent solution and filling to volume:

2.4.1. Remove the volumetric flask or container from the stirring plate in Section 2.3.2 and place it on a bench.

2.4.2. Place a magnetic retriever on the outside of the flask and drag or slide the stir bar out and above the solution level.

2.4.3. Rinse the stir bar with a small quantity of solvent or reagent water as required, into the solution

2.4.4. Completely withdraw the stir bar from the volumetric flask.

2.4.5. Fill the volumetric flask to the mark.

2.4.6. Perform final mixing by repeated inversion.

2.5. Reagent storage:

2.5.1. Transfer the solution from Section 2.4.6 into the recommended storage container. This is given under the individual reagent section.

2.5.2. Label the storage containers following the instruction given in Section 3.

3. Reagent and Standard labels

3.1. All prepared reagents and standards must be stored in appropriately labeled containers.

3.2. Label containers as follow:

3.2.1. Reagent bottles

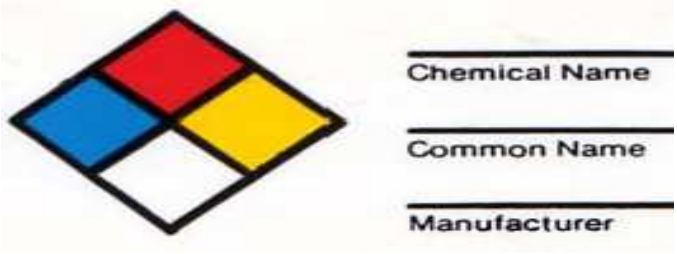
PARAMETER:	(Water Content by Karl Fisher)
IDENTITY:	(_____)
DATE OF PREPARATION:	(mm/dd/yyyy)
EXPIRATION DATE:	(mm/dd/yyyy)
INITIALS OF PREPARER:	(A.A.)
LIMS ID:	_____

3.2.1.1. Excluding reagents that are prepared and discarded daily, all reagents should contain the following label with appropriate chemical information.

3.2.2. Standard bottles

PARAMETER: (Water Content by Karl Fisher)
IDENTITY: (Stock Calibration Standard (SCS))
CONCENTRATION: (1000 mg H_2O/L)
DATE OF PREPARATION: (mm/dd/yyyy)
DATE OF STANDARDIZATION: (mm/dd/yyyy or NA (not applicable))
INITIALS OF PREPARER: (A.A.)
LIMS ID: _____

3.2.2.1. Unless discarded the same day as preparation, all standards should contain the following label with appropriate information.



3.2.3. Commercial reagents used are pre-labeled. Mark date opened.

APPENDIX C – Deviations from Reference Method

Section 6: Method 9000 Revision 0, February 2007 calls for using a water vaporization module (furnace) when interference from hydroxides greater than 1 Normal (N) is suspected. CRL does not have a furnace module. If this interference is suspected the results will be flagged accordingly.

Section 10.5.2: Method 9000 Revision 0, February 2007 calls for performing matrix spikes. This SOP does not perform matrix spikes. CRL has never performed matrix spikes for water content determinations by Karl Fischer titration. Neither is it common practice in Karl Fisher titrations. Therefore, this version of this SOP does not perform matrix spikes. Inclusion of matrix spikes may be warranted for future versions of this SOP after appropriate investigation.